

Trimethylsulfonium 1-amino-6-fluoro-2,3,4,5,7,8,9,10,11,12-decaiodo-1-carba-*closo*-dodecaborate

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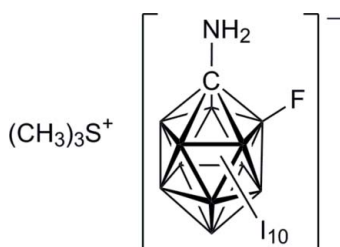
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{B}-\text{B}) = 0.008$ Å; R factor = 0.024; wR factor = 0.052; data-to-parameter ratio = 20.8.

In the asymmetric unit of the title salt, $\text{C}_3\text{H}_9\text{S}^+\cdot\text{CH}_2\text{B}_{11}\text{FI}_{10}\text{N}^-$ or $(\text{CH}_3)_3\text{S}[1\text{-H}_2\text{N-6-F-}closo\text{-1-CB}_{11}\text{I}_{10}]$, both ions lie in general positions. The anion is perfectly ordered and so the positions of the C—NH₂ vertex and the fluorine substituent are clearly assigned. The relatively short C—N bond length may be interpreted in terms of a very electron deficient {*closo*-1-CB₁₁} cluster.

Related literature

For a general overview on monocarba-*closo*-dodecaborates, see: Körbe *et al.* (2006). For the synthesis and properties of 1-aminomonocarba-*closo*-dodecaboron clusters, see: Jelínek *et al.* (1986); Srivastava *et al.* (1996); Finze (2007, 2009); Finze *et al.* (2007); Finze & Sprenger (2010). For studies on the proton affinity of halogenated {*closo*-1-CB₁₁} clusters, see: Himmelspace *et al.* (2011, 2012). For the formation of $(\text{CH}_3)_3\text{S}^+$ from dimethyl sulfoxide, see: Nifontova & Lavrentiev (1993); Forrester *et al.* (1995); Park *et al.* (2005). For the structure of $(\text{CH}_3)_3\text{SBr}$, see: Svensson & Kloo (1996).



Experimental

Crystal data

$\text{C}_3\text{H}_9\text{S}^+\cdot\text{CH}_2\text{B}_{11}\text{FI}_{10}\text{N}^-$
 $M_r = 1512.11$
 Monoclinic, $P2_1/n$
 $a = 10.0672$ (1) Å
 $b = 16.7057$ (2) Å
 $c = 17.5574$ (2) Å
 $\beta = 93.175$ (1)°
 $V = 2948.26$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 10.59$ mm⁻¹
 $T = 100$ K
 $0.79 \times 0.28 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
 Absorption correction: analytical [*CrysAlis PRO* (Oxford Diffraction, 2009), based on expressions derived by Clark & Reid (1995)]
 $T_{\min} = 0.040$, $T_{\max} = 0.204$
 30584 measured reflections
 5482 independent reflections
 5282 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.052$
 $S = 1.31$
 5482 reflections
 264 parameters
 2 restraints
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 1.06$ e Å⁻³
 $\Delta\rho_{\min} = -0.61$ e Å⁻³

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2011); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RU2027).

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supplementary materials

Acta Cryst. (2012). E68, o640 [doi:10.1107/S1600536812004424]

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Comment

Monocarba-*closo*-dodecaborates with amino groups that are bonded to the cluster carbon or boron atoms are potential building blocks for a broad range of applications (Körbe *et al.*, 2006). The properties of the amino group are strongly influenced by (i) the other substituents of the {*closo*-1-CB₁₁} cluster (Jelínek *et al.* 1986; Srivastava *et al.* 1996; Finze *et al.*, 2007; Finze, 2007) and (ii) the type of cluster atom that it is bonded to (Finze, 2009).

Halogenation of all boron vertices of the {*closo*-1-CB₁₁} cluster (Körbe *et al.*, 2006) results in a decrease of the electron density in the cluster. For example, this effect is evident from the calculated proton affinity of [*closo*-1-CB₁₁X₁₁]²⁻ that strongly decreases from X = H to X = halogen (Himmelspach *et al.*, 2012). A further example is the coordination of CH₃CN or H₂O to Hg^{II} of the dianionic mercury(II) complex [Hg(*closo*-1-CB₁₁F₁₁)₂]²⁻, which is related to the Lewis-acidity of mercury (Himmelspach *et al.*, 2011). In contrast, for the complex [Hg(*closo*-1-CB₁₁H₁₁)₂]²⁻ that possess a lower Lewis-acidity at the mercury atom no coordination of a third ligand was observed (Himmelspach *et al.*, 2012). Similarly, a much lower electron density at the amino group is found for halogenated {1-H₂N-*closo*-1-CB₁₁} clusters in comparison to [1-H₂N-*closo*-1-CB₁₁H₁₁]⁻: The non-halogenated anion is easily protonated to yield 1-H₃N-*closo*-1-CB₁₁H₁₁ (pK_a = 6.0) (Jelínek *et al.* 1986; Finze, 2009) while the attempted protonation of [1-H₂N-6-F-*closo*-1-CB₁₁I₁₀]⁻ with conc. hydrochloric acid failed (Finze & Sprenger, 2010).

Treatment of a solution of H(solv)[1-H₂N-6-F-*closo*-1-CB₁₁I₁₀] in dimethyl sulfoxide (DMSO) with methanol and conc. hydrochloric acid results in the slow formation of crystals of the trimethylsulfonium salt Me₃S[1-H₂N-6-F-*closo*-1-CB₁₁I₁₀]. Similar reactions of DMSO to result in trimethylsulfonium salts were reported earlier (*e.g.* Nifontova & Lavrentiev, 1993; Forrester *et al.*, 1995; Park *et al.*, 2005).

The title compound trimethylsulfonium-1-amino-6-fluoro-1-carba-*closo*-dodecaborate (Figure 1) crystallizes in the monoclinic space group *P*2₁/*n* with one formula unit in the asymmetric unit. The position of the C–NH₂ and the B–F vertex of the [1-H₂N-6-F-*closo*-1-CB₁₁I₁₀]⁻ anion are clearly assigned (i) from comparative refinements and (ii) the C–N and B–F as well as the inner-cluster C–B bond lengths are similar to values reported for [1-H₂N-*closo*-1-CB₁₁F₁₁]⁻ and [1-H₂N-6-HO-*closo*-1-CB₁₁F₁₀]⁻ (Finze *et al.*, 2007). The bond lengths and angles of the trimethylsulfonium cation are similar to those reported for other (CH₃)₃S⁺ salts, for example (CH₃)₃SBr (Svensson & Kloo, 1996).

Experimental

[Et₄N][1-H₂N-6-F-*closo*-1-CB₁₁I₁₀] (50 mg, 0.03 mmol), which was synthesized according to a published procedure (Finze & Sprenger, 2010), was suspended in a mixture of aqueous HCl (50 ml, 10% v/v) and diethyl ether (50 ml). After 30 minutes of stirring the solid dissolved. The ethereal layer was separated and the aqueous solution was extracted with Et₂O (2 x 20 ml). The combined ether phases were dried with MgSO₄, filtered, and most of the diethyl ether was removed under reduced pressure. DMSO was added (2 ml) and the residual Et₂O was removed under reduced pressure. Methanol

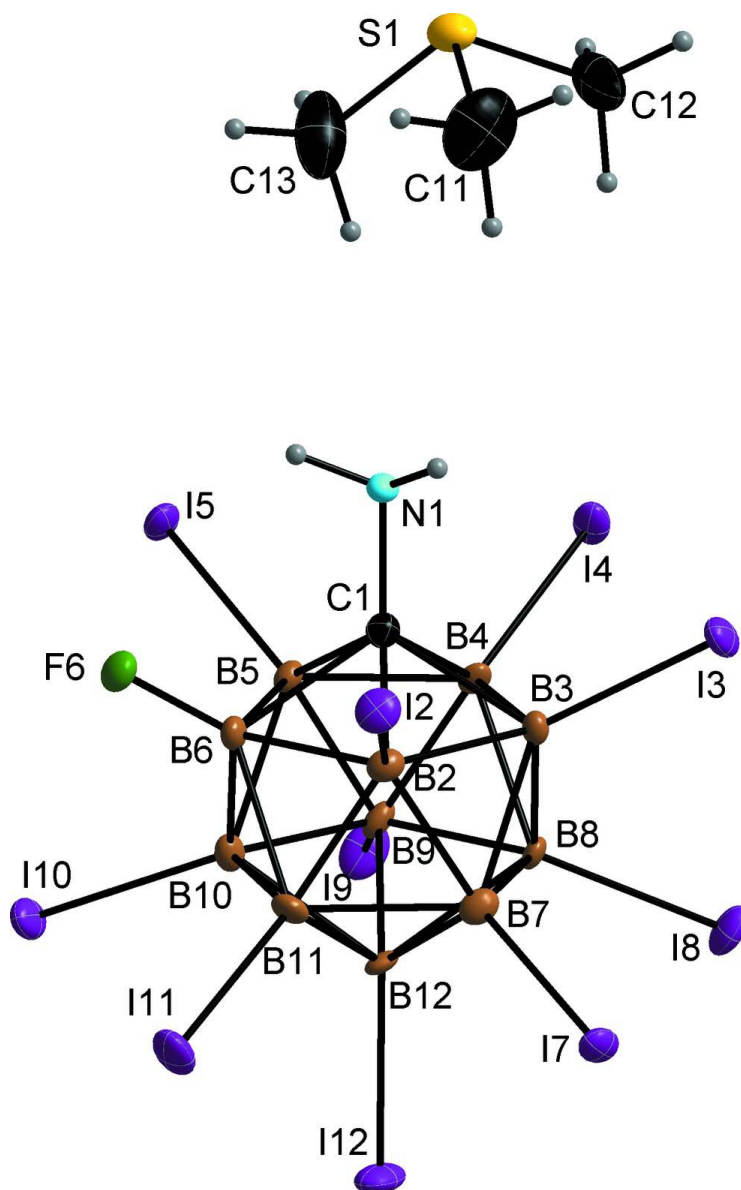
(2 ml) and conc. hydrochloric acid (4 ml) was added. According to $^{11}\text{B}\{^1\text{H}\}$ -NMR spectroscopy the $[1\text{-H}_2\text{N-6-F-closo-1-CB}_{11}\text{I}_{10}]^-$ anion was not protonated. After 3 month crystals of the title compound were obtained from the reaction mixture. ^1H NMR (500.13 MHz, CD_2Cl_2): $d = 2.90$ p.p.m. (s, $(\text{CH}_3)_3\text{S}^+$) (the signal of the protons of the amino group was not observed as a result of signal broadening). ^{11}B NMR (160.46 MHz, CD_2Cl_2): $d = -1.6$ (s, 1 B, BF, B-2), -14.3 (s, 1 B, B-12), -17.1 (s, 2 B, B-4 and B-5), -18.9 (s, 2 B, B-8 and B-10), -18.9 (s, 2 B, B-3 and B-6), -19.2 (s, 2 B, B-7 and B-11), -21.0 (s, 1 B, B-9). IR (ATR): $\nu_{\text{as}}(\text{NH}_2)$ 3362 *versus*, $\nu_{\text{s}}(\text{NH}_2)$ 3297 cm^{-1} *versus*.

Refinement

All hydrogen atoms of the CH_3 groups were refined using a riding model with the $U_{\text{iso}}(\text{H})$ set to $1.5U_{\text{eq}}(\text{C})$. The coordinates of the two hydrogen atoms at the nitrogen atom were refined unrestrictedly together with one refined common U_{iso} value. Anisotropic displacement parameters of all non-hydrogen atoms were also refined unrestrictedly.

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).


Figure 1

The structure of the asymmetric unit, showing 50% probability displacement ellipsoids. Hydrogen atoms are shown as spheres of arbitrary radius.

Trimethylsulfonium 1-amino-6-fluoro-2,3,4,5,7,8,9,10,11,12-decaiodo- 1-carba-*closo*-dodecaborate
Crystal data
 $\text{C}_3\text{H}_9\text{S}^+\cdot\text{CH}_2\text{B}_{11}\text{FI}_{10}\text{N}^-$
 $M_r = 1512.11$

 Monoclinic, $P2_1/n$

 Hall symbol: $-P\ 2_1/n$
 $a = 10.0672\ (1)\ \text{\AA}$
 $b = 16.7057\ (2)\ \text{\AA}$
 $c = 17.5574\ (2)\ \text{\AA}$
 $\beta = 93.175\ (1)^\circ$
 $V = 2948.26\ (6)\ \text{\AA}^3$
 $Z = 4$
 $F(000) = 2608$
 $D_x = 3.407\ \text{Mg m}^{-3}$

 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 35017 reflections

 $\theta = 3.0\text{--}34.0^\circ$
 $\mu = 10.59\ \text{mm}^{-1}$

$T = 100$ K $0.79 \times 0.28 \times 0.20$ mm
 Block, colourless

Data collection

| | |
|--|--|
| Oxford Diffraction Xcalibur Eos diffractometer | $T_{\min} = 0.040$, $T_{\max} = 0.204$ |
| Radiation source: fine-focus sealed tube | 30584 measured reflections |
| Equatorial mounted graphite monochromator | 5482 independent reflections |
| Detector resolution: 16.2711 pixels mm ⁻¹ | 5282 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.029$ |
| Absorption correction: analytical [CrysAlis PRO (Oxford Diffraction, 2009), based on expressions derived by Clark & Reid (1995)] | $\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 3.2^\circ$ |
| | $h = -12 \rightarrow 12$ |
| | $k = -20 \rightarrow 20$ |
| | $l = -21 \rightarrow 21$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.024$ | $w = 1/[\sigma^2(F_o^2) + (0.014P)^2 + 16.P]$ |
| $wR(F^2) = 0.052$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.31$ | $(\Delta/\sigma)_{\max} = 0.001$ |
| 5482 reflections | $\Delta\rho_{\max} = 1.06 \text{ e } \text{\AA}^{-3}$ |
| 264 parameters | $\Delta\rho_{\min} = -0.61 \text{ e } \text{\AA}^{-3}$ |
| 2 restraints | Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.000548 (19) |
| Secondary atom site location: difference Fourier map | |

Special details

Experimental. CrysAlisPro, Oxford Diffraction (2009). Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995).

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|---------------|----------------------------------|
| C1 | 0.2112 (5) | 0.3730 (3) | 0.5075 (3) | 0.0111 (10) |
| N1 | 0.2183 (4) | 0.4462 (3) | 0.4682 (2) | 0.0107 (9) |
| H12 | 0.292 (4) | 0.475 (4) | 0.480 (4) | 0.034 (14)* |
| H11 | 0.148 (5) | 0.478 (4) | 0.475 (4) | 0.034 (14)* |
| B2 | 0.2708 (6) | 0.3639 (4) | 0.6030 (3) | 0.0105 (11) |
| I2 | 0.34637 (3) | 0.46793 (2) | 0.661845 (19) | 0.01491 (9) |
| B3 | 0.3549 (6) | 0.3180 (4) | 0.5266 (3) | 0.0087 (11) |
| I3 | 0.54383 (3) | 0.36148 (2) | 0.494809 (19) | 0.01425 (9) |
| B4 | 0.2327 (6) | 0.2837 (4) | 0.4561 (3) | 0.0090 (11) |
| I4 | 0.27883 (3) | 0.28803 (2) | 0.338027 (18) | 0.01482 (9) |

| | | | | |
|------|--------------|--------------|---------------|-------------|
| B5 | 0.0752 (6) | 0.3128 (3) | 0.4868 (3) | 0.0088 (11) |
| I5 | -0.08352 (3) | 0.35589 (2) | 0.411424 (19) | 0.01391 (8) |
| B6 | 0.0985 (6) | 0.3608 (4) | 0.5781 (3) | 0.0094 (11) |
| F6 | 0.0191 (3) | 0.42395 (19) | 0.59388 (17) | 0.0160 (7) |
| B7 | 0.3288 (6) | 0.2642 (4) | 0.6136 (3) | 0.0112 (12) |
| I7 | 0.49137 (3) | 0.23918 (2) | 0.694333 (18) | 0.01296 (8) |
| B8 | 0.3054 (6) | 0.2145 (4) | 0.5231 (3) | 0.0092 (11) |
| I8 | 0.44339 (4) | 0.12326 (2) | 0.49050 (2) | 0.01707 (9) |
| B9 | 0.1296 (6) | 0.2120 (4) | 0.4979 (3) | 0.0105 (11) |
| I9 | 0.03159 (4) | 0.11836 (2) | 0.43173 (2) | 0.02016 (9) |
| B10 | 0.0465 (6) | 0.2599 (4) | 0.5729 (3) | 0.0105 (11) |
| I10 | -0.15349 (3) | 0.23210 (2) | 0.60119 (2) | 0.01709 (9) |
| B11 | 0.1677 (6) | 0.2908 (4) | 0.6452 (3) | 0.0104 (11) |
| I11 | 0.12763 (4) | 0.29664 (2) | 0.763929 (19) | 0.01921 (9) |
| B12 | 0.1890 (6) | 0.1985 (3) | 0.5955 (3) | 0.0089 (11) |
| I12 | 0.17344 (4) | 0.08517 (2) | 0.65238 (2) | 0.01909 (9) |
| S1 | 0.16683 (16) | 0.54368 (9) | 0.20914 (9) | 0.0228 (3) |
| C11 | 0.2577 (8) | 0.5579 (5) | 0.2974 (5) | 0.044 (2) |
| H111 | 0.2125 | 0.5962 | 0.3275 | 0.066* |
| H112 | 0.3452 | 0.5773 | 0.2883 | 0.066* |
| H113 | 0.2647 | 0.5079 | 0.3243 | 0.066* |
| C12 | 0.2548 (8) | 0.4642 (4) | 0.1672 (4) | 0.0377 (18) |
| H121 | 0.3398 | 0.4833 | 0.1528 | 0.057* |
| H122 | 0.2047 | 0.4450 | 0.1228 | 0.057* |
| H123 | 0.2675 | 0.4214 | 0.2034 | 0.057* |
| C13 | 0.0275 (7) | 0.4896 (4) | 0.2388 (5) | 0.0403 (19) |
| H131 | 0.0574 | 0.4445 | 0.2689 | 0.060* |
| H132 | -0.0246 | 0.4712 | 0.1948 | 0.060* |
| H133 | -0.0258 | 0.5238 | 0.2687 | 0.060* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|--------------|--------------|--------------|---------------|---------------|---------------|
| C1 | 0.011 (3) | 0.011 (3) | 0.011 (2) | 0.001 (2) | 0.001 (2) | 0.000 (2) |
| N1 | 0.009 (2) | 0.010 (2) | 0.013 (2) | 0.0001 (17) | 0.0016 (17) | 0.0037 (18) |
| B2 | 0.012 (3) | 0.009 (3) | 0.011 (3) | -0.001 (2) | -0.001 (2) | 0.000 (2) |
| I2 | 0.01739 (18) | 0.01209 (18) | 0.01497 (17) | -0.00177 (13) | -0.00148 (13) | -0.00448 (13) |
| B3 | 0.007 (3) | 0.011 (3) | 0.008 (3) | 0.001 (2) | 0.001 (2) | -0.001 (2) |
| I3 | 0.00892 (16) | 0.01894 (19) | 0.01509 (17) | -0.00289 (13) | 0.00248 (13) | 0.00286 (14) |
| B4 | 0.010 (3) | 0.012 (3) | 0.005 (2) | 0.003 (2) | -0.002 (2) | 0.000 (2) |
| I4 | 0.01385 (17) | 0.0234 (2) | 0.00719 (16) | 0.00384 (14) | 0.00041 (12) | -0.00055 (13) |
| B5 | 0.008 (3) | 0.008 (3) | 0.011 (3) | 0.001 (2) | -0.001 (2) | 0.000 (2) |
| I5 | 0.01020 (17) | 0.01890 (19) | 0.01234 (16) | 0.00449 (13) | -0.00204 (13) | 0.00323 (13) |
| B6 | 0.006 (3) | 0.012 (3) | 0.011 (3) | 0.001 (2) | 0.001 (2) | 0.001 (2) |
| F6 | 0.0159 (16) | 0.0151 (16) | 0.0170 (16) | 0.0052 (13) | 0.0020 (12) | -0.0007 (13) |
| B7 | 0.012 (3) | 0.010 (3) | 0.012 (3) | 0.000 (2) | -0.003 (2) | -0.001 (2) |
| I7 | 0.01068 (17) | 0.01716 (19) | 0.01059 (16) | 0.00013 (13) | -0.00340 (12) | 0.00326 (13) |
| B8 | 0.007 (3) | 0.010 (3) | 0.011 (3) | 0.004 (2) | 0.000 (2) | 0.001 (2) |
| I8 | 0.01617 (18) | 0.01587 (19) | 0.01883 (18) | 0.00825 (14) | -0.00215 (14) | -0.00469 (14) |
| B9 | 0.008 (3) | 0.010 (3) | 0.013 (3) | 0.003 (2) | -0.003 (2) | -0.001 (2) |

| | | | | | | |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| I9 | 0.01760 (19) | 0.01479 (19) | 0.0273 (2) | -0.00160 (14) | -0.00620 (15) | -0.00826 (15) |
| B10 | 0.007 (3) | 0.015 (3) | 0.010 (3) | 0.000 (2) | 0.000 (2) | 0.000 (2) |
| I10 | 0.00822 (17) | 0.0216 (2) | 0.02161 (18) | -0.00206 (14) | 0.00219 (13) | 0.00644 (15) |
| B11 | 0.013 (3) | 0.013 (3) | 0.006 (3) | -0.001 (2) | 0.002 (2) | 0.002 (2) |
| I11 | 0.01868 (19) | 0.0299 (2) | 0.00953 (16) | 0.00059 (15) | 0.00519 (13) | 0.00235 (15) |
| B12 | 0.008 (3) | 0.006 (3) | 0.012 (3) | 0.002 (2) | -0.001 (2) | 0.004 (2) |
| I12 | 0.01749 (18) | 0.01344 (19) | 0.0260 (2) | -0.00212 (14) | -0.00231 (15) | 0.01093 (15) |
| S1 | 0.0301 (8) | 0.0173 (7) | 0.0215 (7) | 0.0052 (6) | 0.0055 (6) | 0.0044 (6) |
| C11 | 0.034 (4) | 0.044 (5) | 0.052 (5) | -0.004 (3) | -0.017 (4) | -0.013 (4) |
| C12 | 0.051 (5) | 0.022 (4) | 0.044 (4) | 0.011 (3) | 0.032 (4) | 0.003 (3) |
| C13 | 0.024 (4) | 0.030 (4) | 0.067 (5) | -0.007 (3) | 0.011 (3) | -0.020 (4) |

Geometric parameters (Å, °)

| | | | |
|----------|------------|-----------|-----------|
| C1—N1 | 1.409 (7) | B7—B8 | 1.796 (8) |
| C1—B5 | 1.721 (8) | B7—B12 | 1.799 (8) |
| C1—B3 | 1.730 (7) | B7—B11 | 1.798 (8) |
| C1—B6 | 1.738 (7) | B7—I7 | 2.146 (6) |
| C1—B2 | 1.756 (8) | B8—B12 | 1.796 (8) |
| C1—B4 | 1.764 (8) | B8—B9 | 1.800 (8) |
| N1—H12 | 0.898 (10) | B8—I8 | 2.161 (6) |
| N1—H11 | 0.896 (10) | B9—B10 | 1.788 (8) |
| B2—B6 | 1.767 (8) | B9—B12 | 1.797 (8) |
| B2—B7 | 1.772 (8) | B9—I9 | 2.155 (6) |
| B2—B11 | 1.788 (8) | B10—B11 | 1.787 (8) |
| B2—B3 | 1.797 (8) | B10—B12 | 1.791 (8) |
| B2—I2 | 2.140 (6) | B10—I10 | 2.151 (6) |
| B3—B4 | 1.791 (8) | B11—B12 | 1.791 (8) |
| B3—B8 | 1.799 (8) | B11—I11 | 2.148 (6) |
| B3—B7 | 1.804 (8) | B12—I12 | 2.150 (6) |
| B3—I3 | 2.139 (6) | S1—C13 | 1.770 (7) |
| B4—B9 | 1.771 (8) | S1—C11 | 1.771 (7) |
| B4—B5 | 1.771 (8) | S1—C12 | 1.778 (6) |
| B4—B8 | 1.778 (8) | C11—H111 | 0.9600 |
| B4—I4 | 2.150 (5) | C11—H112 | 0.9600 |
| B5—B9 | 1.778 (8) | C11—H113 | 0.9600 |
| B5—B10 | 1.787 (8) | C12—H121 | 0.9600 |
| B5—B6 | 1.795 (8) | C12—H122 | 0.9600 |
| B5—I5 | 2.143 (6) | C12—H123 | 0.9600 |
| B6—F6 | 1.361 (7) | C13—H131 | 0.9600 |
| B6—B10 | 1.765 (8) | C13—H132 | 0.9600 |
| B6—B11 | 1.774 (8) | C13—H133 | 0.9600 |
| N1—C1—B5 | 117.7 (4) | B11—B7—B3 | 108.3 (4) |
| N1—C1—B3 | 119.5 (4) | B2—B7—I7 | 119.1 (4) |
| B5—C1—B3 | 112.1 (4) | B8—B7—I7 | 123.5 (4) |
| N1—C1—B6 | 120.2 (4) | B12—B7—I7 | 124.0 (4) |
| B5—C1—B6 | 62.5 (3) | B11—B7—I7 | 120.8 (3) |
| B3—C1—B6 | 111.8 (4) | B3—B7—I7 | 121.0 (4) |
| N1—C1—B2 | 121.3 (4) | B4—B8—B12 | 107.7 (4) |

| | | | |
|------------|-----------|-------------|-----------|
| B5—C1—B2 | 112.1 (4) | B4—B8—B7 | 108.3 (4) |
| B3—C1—B2 | 62.1 (3) | B12—B8—B7 | 60.1 (3) |
| B6—C1—B2 | 60.7 (3) | B4—B8—B3 | 60.1 (3) |
| N1—C1—B4 | 118.3 (4) | B12—B8—B3 | 108.0 (4) |
| B5—C1—B4 | 61.1 (3) | B7—B8—B3 | 60.2 (3) |
| B3—C1—B4 | 61.6 (3) | B4—B8—B9 | 59.3 (3) |
| B6—C1—B4 | 111.7 (4) | B12—B8—B9 | 59.9 (3) |
| B2—C1—B4 | 111.8 (4) | B7—B8—B9 | 107.9 (4) |
| C1—N1—H12 | 114 (5) | B3—B8—B9 | 107.3 (4) |
| C1—N1—H11 | 113 (5) | B4—B8—I8 | 122.0 (3) |
| H12—N1—H11 | 108 (7) | B12—B8—I8 | 122.5 (3) |
| C1—B2—B6 | 59.1 (3) | B7—B8—I8 | 120.3 (3) |
| C1—B2—B7 | 105.9 (4) | B3—B8—I8 | 120.4 (3) |
| B6—B2—B7 | 108.2 (4) | B9—B8—I8 | 123.8 (4) |
| C1—B2—B11 | 106.2 (4) | B4—B9—B5 | 59.9 (3) |
| B6—B2—B11 | 59.9 (3) | B4—B9—B10 | 108.2 (4) |
| B7—B2—B11 | 60.7 (3) | B5—B9—B10 | 60.2 (3) |
| C1—B2—B3 | 58.3 (3) | B4—B9—B12 | 108.0 (4) |
| B6—B2—B3 | 107.4 (4) | B5—B9—B12 | 107.9 (4) |
| B7—B2—B3 | 60.7 (3) | B10—B9—B12 | 59.9 (3) |
| B11—B2—B3 | 109.0 (4) | B4—B9—B8 | 59.7 (3) |
| C1—B2—I2 | 119.0 (3) | B5—B9—B8 | 107.3 (4) |
| B6—B2—I2 | 117.5 (4) | B10—B9—B8 | 107.6 (4) |
| B7—B2—I2 | 127.4 (4) | B12—B9—B8 | 59.9 (3) |
| B11—B2—I2 | 123.6 (4) | B4—B9—I9 | 121.9 (4) |
| B3—B2—I2 | 122.5 (4) | B5—B9—I9 | 119.9 (3) |
| C1—B3—B4 | 60.1 (3) | B10—B9—I9 | 120.2 (4) |
| C1—B3—B2 | 59.7 (3) | B12—B9—I9 | 122.8 (4) |
| B4—B3—B2 | 108.6 (4) | B8—B9—I9 | 124.3 (4) |
| C1—B3—B8 | 106.1 (4) | B6—B10—B11 | 59.9 (3) |
| B4—B3—B8 | 59.4 (3) | B6—B10—B5 | 60.7 (3) |
| B2—B3—B8 | 107.1 (4) | B11—B10—B5 | 108.7 (4) |
| C1—B3—B7 | 105.6 (4) | B6—B10—B9 | 108.4 (4) |
| B4—B3—B7 | 107.4 (4) | B11—B10—B9 | 108.8 (4) |
| B2—B3—B7 | 58.9 (3) | B5—B10—B9 | 59.6 (3) |
| B8—B3—B7 | 59.8 (3) | B6—B10—B12 | 107.7 (4) |
| C1—B3—I3 | 121.0 (4) | B11—B10—B12 | 60.1 (3) |
| B4—B3—I3 | 121.0 (3) | B5—B10—B12 | 107.8 (4) |
| B2—B3—I3 | 120.8 (3) | B9—B10—B12 | 60.3 (3) |
| B8—B3—I3 | 124.5 (3) | B6—B10—I10 | 118.2 (3) |
| B7—B3—I3 | 124.3 (3) | B11—B10—I10 | 120.6 (3) |
| C1—B4—B9 | 105.6 (4) | B5—B10—I10 | 120.0 (3) |
| C1—B4—B5 | 58.3 (3) | B9—B10—I10 | 123.8 (4) |
| B9—B4—B5 | 60.3 (3) | B12—B10—I10 | 125.0 (4) |
| C1—B4—B8 | 105.6 (4) | B6—B11—B10 | 59.4 (3) |
| B9—B4—B8 | 61.0 (3) | B6—B11—B12 | 107.3 (4) |
| B5—B4—B8 | 108.6 (4) | B10—B11—B12 | 60.1 (3) |
| C1—B4—B3 | 58.3 (3) | B6—B11—B2 | 59.5 (3) |
| B9—B4—B3 | 109.0 (4) | B10—B11—B2 | 107.0 (4) |

| | | | |
|------------|-----------|---------------|-----------|
| B5—B4—B3 | 107.1 (4) | B12—B11—B2 | 107.4 (4) |
| B8—B4—B3 | 60.6 (3) | B6—B11—B7 | 106.7 (4) |
| C1—B4—I4 | 120.3 (3) | B10—B11—B7 | 107.6 (4) |
| B9—B4—I4 | 125.9 (4) | B12—B11—B7 | 60.2 (3) |
| B5—B4—I4 | 122.0 (3) | B2—B11—B7 | 59.2 (3) |
| B8—B4—I4 | 123.9 (3) | B6—B11—I11 | 121.8 (4) |
| B3—B4—I4 | 118.6 (3) | B10—B11—I11 | 123.0 (3) |
| C1—B5—B4 | 60.6 (3) | B12—B11—I11 | 123.1 (4) |
| C1—B5—B9 | 107.1 (4) | B2—B11—I11 | 121.0 (4) |
| B4—B5—B9 | 59.9 (3) | B7—B11—I11 | 122.1 (3) |
| C1—B5—B10 | 105.9 (4) | B11—B12—B10 | 59.9 (3) |
| B4—B5—B10 | 108.2 (4) | B11—B12—B8 | 108.5 (4) |
| B9—B5—B10 | 60.2 (3) | B10—B12—B8 | 107.7 (4) |
| C1—B5—B6 | 59.2 (3) | B11—B12—B9 | 108.3 (4) |
| B4—B5—B6 | 108.7 (4) | B10—B12—B9 | 59.8 (3) |
| B9—B5—B6 | 107.5 (4) | B8—B12—B9 | 60.2 (3) |
| B10—B5—B6 | 59.0 (3) | B11—B12—B7 | 60.1 (3) |
| C1—B5—I5 | 119.4 (3) | B10—B12—B7 | 107.4 (4) |
| B4—B5—I5 | 123.6 (3) | B8—B12—B7 | 60.0 (3) |
| B9—B5—I5 | 126.9 (4) | B9—B12—B7 | 107.9 (4) |
| B10—B5—I5 | 122.6 (3) | B11—B12—I12 | 121.2 (3) |
| B6—B5—I5 | 117.2 (3) | B10—B12—I12 | 122.0 (3) |
| F6—B6—C1 | 117.9 (4) | B8—B12—I12 | 121.8 (3) |
| F6—B6—B10 | 125.1 (4) | B9—B12—I12 | 121.6 (4) |
| C1—B6—B10 | 106.2 (4) | B7—B12—I12 | 122.1 (3) |
| F6—B6—B2 | 120.4 (5) | C13—S1—C11 | 101.0 (4) |
| C1—B6—B2 | 60.1 (3) | C13—S1—C12 | 99.3 (3) |
| B10—B6—B2 | 109.0 (4) | C11—S1—C12 | 102.5 (4) |
| F6—B6—B11 | 126.1 (4) | S1—C11—H111 | 109.5 |
| C1—B6—B11 | 107.6 (4) | S1—C11—H112 | 109.5 |
| B10—B6—B11 | 60.7 (3) | H111—C11—H112 | 109.5 |
| B2—B6—B11 | 60.7 (3) | S1—C11—H113 | 109.5 |
| F6—B6—B5 | 118.6 (4) | H111—C11—H113 | 109.5 |
| C1—B6—B5 | 58.3 (3) | H112—C11—H113 | 109.5 |
| B10—B6—B5 | 60.3 (3) | S1—C12—H121 | 109.5 |
| B2—B6—B5 | 108.2 (4) | S1—C12—H122 | 109.5 |
| B11—B6—B5 | 109.0 (4) | H121—C12—H122 | 109.5 |
| B2—B7—B8 | 108.4 (4) | S1—C12—H123 | 109.5 |
| B2—B7—B12 | 107.7 (4) | H121—C12—H123 | 109.5 |
| B8—B7—B12 | 59.9 (3) | H122—C12—H123 | 109.5 |
| B2—B7—B11 | 60.1 (3) | S1—C13—H131 | 109.5 |
| B8—B7—B11 | 108.1 (4) | S1—C13—H132 | 109.5 |
| B12—B7—B11 | 59.7 (3) | H131—C13—H132 | 109.5 |
| B2—B7—B3 | 60.4 (3) | S1—C13—H133 | 109.5 |
| B8—B7—B3 | 60.0 (3) | H131—C13—H133 | 109.5 |
| B12—B7—B3 | 107.7 (4) | H132—C13—H133 | 109.5 |